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VOLATILISATION OF PESTICIDES FROM SOIL AND PLANT SURFACES

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Dedicated to Prof. Dr. Werner Klein on the occasion of his 60th birthday

ABSTRACT

Volatilisation rates of five pesticides (parathion-methyl, endosulfan, fenpropimorph, lindane, trifluralin) applied in formulations were determined from soil surfaces and French beans. Tests were performed in a wind tunnel under defined conditions (20°C; air velocity 1 m/s; relative air humidity 40 - 60 %). Volatilised substances in the air were trapped by passing a part of the whole air stream of the wind tunnel through polyurethane foam. Toluene extracts of the adsorbent were analyzed by gas chromatography. Volatilisation from soil surfaces within 24 hours increased in the order parathion-methyl = fenpropimorph < endosulfan < lindane < trifluralin. For all compounds volatilisation from French beans was higher as compared to volatilisation from soil surfaces. Volatilisation rates from plant surfaces were 50 - 70 % for parathion-methyl, endosulfan, fenpropimorph, and lindane. Trifluralin was nearly completely volatilised within 24 hours. Volatilisation and vapour pressure seemed to be correlated for the tested pesticides.
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Key words: pesticides, volatilisation, wind tunnel, air sampling, endosulfan, fenpropimorph, lindane, parathion-methyl, trifluralin

INTRODUCTION

Volatilisation may be an important factor for the dissipation of pesticides in the environment. In Germany, in the last years the Federal Biological Agency (Biologische Bundesanstalt, BBA) has required data on the volatilisation potential of active ingredients (a.i.) of pesticides for risk

assessment within the registration process. Therefore, new interest has been focused on volatilisation experiments which are performed under field relevant conditions. Ideal for this studies are model chamber systems (e.g., wind tunnels) where climatic parameters are controlable.

The experimental requirements for the performance of volatilisation studies with pesticides for registration purposes are laid down in a BBA-guideline [1]. The following standard parameters are required for model chamber investigations: air velocity $\geq$ 1 m/s, temperature 20°C, relative air humidity 35 %. Since the air humidity value is relatively low and not realistic for field situations, this condition has become obsolete [2]. Mostly, a more realistic value of approximately 50 % relative humidity is used.

The BBA-guideline [1] demands experimental conditions for volatilisation measurements to be as close as possible to field conditions. Therefore, pesticide treatments of soil or plant surfaces should be performed using a moving nozzle which is also licensed for field applications. As in the field, aqueous preparations of the formulated products are applied.

According to the guideline, the volatilisation rate has to be calculated in relation to the amount of pesticide applied. For pesticides with volatilisation rates below 20 %, volatilisation is not considered as critical. If the volatilisation rate is above this threshold, further investigations would be necessary (e.g., an assessment of the atmospheric oxidation potential [1]).

A wind tunnel system [3] was used for the investigation of the volatility of five pesticides (parathion-methyl, endosulfan, fenpropimorph, lindane, and trifluralin) under the conditions of the BBA-guideline [1]. The results obtained should help to understand the process of volatilisation and the factors controlling it. Of special interest was the question whether a correlation between the volatilisation and the vapour pressure exists.

MATERIALS AND METHODS

Model substances

Parathion-methyl (organophosphorus insecticide): CAS RN: 298-00-0; IUPAC name: 0,0-dimethyl-0-4-nitrophenyl-phosphorothioate; origin of the formulation: Bayer AG; a.i.-content of the formulation: 400 g/l; emulsion concentrate (EC); actual application doses 25 - 40 mg/m^2.

Endosulfan (organochlorine insecticide): CAS RN: 115-29-7; IUPAC-name: (1,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylenebismethylene) sulfite; origin of the formulation: Hoechst AG (Thiodan EC35); a.i.-content of the formulation: 352 g/l; emulsion concentrate (EC); actual application doses 61 and 56 mg/m^2. The formulation contains a 70:30 mixture of the two isomers alpha-endosulfan and beta-endosulfan. Data given here are the sums of both isomers.

Fenpropimorph (morpholine fungicide): CAS RN: 67564-91-4; IUPAC-name: ($\pm$)-cis-4-[3-[4-tert.-butyl-phenyl)]-2-methylpropyl]-2,6-dimethylmorpholine; origin of the formulation: Spiess-Urania

(Corbel); a.i.-content of the formulation: 750 g/l; emulsion concentrate (EC); actual application doses 70 - 98 mg/m^2.

Lindane (organochlorine insecticide): CAS RN: 58-89-9; IUPAC name: 1,2,4,5/3,6-gamma-hexa-chlorocyclohexane; origin of the formulation: Shell Agrar GmbH (Nexit flüssig SC); a.i.-content of the formulation: 800 g/l; suspension concentrate (SC); actual application doses 112 - 156 mg/m^2.

Trifluralin (2,6-dinitroaniline herbicide): CAS RN: 1582-09-8; IUPAC name: alpha, alpha, alpha-trifluoro-2,6-dinitro-N,N-dipropyl-p-toluidine; origin of the formulation: Zera GmbH (Zera-Trifluralin); a.i.-content of the formulation: 480 g/l; emulsion concentrate (EC); actual application doses 68 and 40 mg/m^2.

Wind tunnel system and air sampling

A detailed description of the wind tunnel system, the air sampling system, and the performance of the experiments was published by Rüdel und Waymann [3]. In principle, a climatised air stream was passed over a target surface of soil or plants (approximately 3000 m^3/h; the actual volume stream was determined within each test).

The volatilisation was determined directly by analysing the a.i.-content of the air stream. Therefore, a portion (2 - 3 %) of the air stream was sucked through two polyurethane foam (PUF) plugs. The plugs were 10 cm in height and diameter, respectively. Extractions were performed with toluene (three times). Extracts were combined, concentrated by evaporation, and analysed by gas chromatography (GC). For all tests, the second PUF plug contained only traces of volatilised compounds (no break through).

Soil tests

A sieved silty sand soil was used (75 - 79 % sand, 1 - 1.5 % organic carbon content; origin: Neustadt a.R., FRG); a 3 cm-layer of the soil was kept at approximately 60 % of the maximum water holding capacity by underlaying with a layer of wet burnt clay granules in stainless steel bowls with 0.14 m^2 surface area. Usually, for each test four soil bowls were used. However, for three of the tests two or six bowls were applied.

Plant tests

French beans (Phaseolus vulgaris) were grown in a climate chamber for four to eight weeks (growth stage: blooming or first fruit bearing; height 30 - 40 cm). Eight bean plants were planted in each stainless steel bowl of 0.09 m^2 surface area. At the end of the volatilisation test (after 24 hours), the plants were rinsed with methanol and the residues in the solution were determined by GC. For each plant test, four bowls planted with beans were used.

Applications

Applications were performed as described by Waymann and Rüdel [4] using an application device with a moving nozzle for spraying the application solution onto the target area. The application device was separate from the wind tunnel. Within 5 - 10 min after each application, the treated bowls were transferred into the volatilisation chamber of the wind tunnel.

For the soil tests, the application dose on the target area was calculated as difference: total amount sprayed minus application losses (residues beside the target area). The application dose for the plant tests was calculated as sum of recovered residues: amount of a.i volatilised plus amount of a.i. rinsed from the plants. During the plant application, the soil below the plants was covered with absorbent paper which was removed before transferring the bowls into the wind tunnel.

Analytical methods

The target compounds were analysed by capillary GC and nitrogen-phosphorus detection or electron-capture detection. The limits of detection for each of the four sampling intervals of each test were: 5 µg/l for parathion-methyl (corresponding to 0.5 - 1 % parathion-methyl volatilised), 10 µg/l test solution for endosulfan (sum of both isomers; corresponding to approximately 1 % endosulfan volatilised), 60 µg/l for fenpropimorph (corresponding to 3 - 4 % fenpropimorph volatilised), 5 µg/l lindane (corresponding to approximately 1 % lindane volatilised), and 0.1 µg/l trifluralin (corresponding to approximately 1 % trifluralin volatilised). Detailed descriptions of the analytical methods are given by von Triller [5], Kördel et al. [6], Waymann and Rüdel [4], and Grass et al. [7].

RESULTS AND DISCUSSION

As model substances pesticides were chosen which were suspected to have a high volatilisation rate (parathion-methyl, endosulfan, fenpropimorph, lindane, and trifluralin). Therefore, the data set is not representative. Furthermore, soil and French bean treatments are not regular application targets for some of the pesticides (e.g., trifluralin usually is not applied on plants; insecticides are not applied onto bare soil). Additionally, the application doses used for these model experiments may differ from the recommendations of the manufacturer.

The volatilisation data of 13 volatilisation tests from soil and plant surfaces are listed in table 1. During all experiments the averaged climate conditions were within the following ranges: air temperature, 19 - 21°C; air velocity, 1 - 1.1 m/s; relative air humidity, 40 - 60 %.

Volatilisation from soil surfaces within 24 hours increased in the order parathion methyl = fenpropimorph < endosulfan < lindane < trifluralin. Usually, four soil bowls per soil test were used. For lindane, soil tests were performed with two and six bowls per test, respectively. In order to compare the soil volatilisation of lindane with other volatilisation data it is assumed that

the volatilisation for four bowls would be the mean of the two tests with two and six bowls (27 % volatilisation within 24 h).

Table 1:   Results from the volatilisation tests with parathion-methyl, endosulfan, fenpropimorph, lindane, and trifluralin.
Data from plant and soil tests. For the soil tests no mass balance could be determined since only air samplings were performed. If not otherwise noted, four bowls were used for each test and the initial amount on the target area was calculated as difference of the amount applied minus the application losses.

Test substance	Volatilisation in % of application dose					Total Recovery
	0 - 1 h	1 - 3 h	3 - 6 h	6 - 24 h	0 - 24 h	
soil tests:						
parathion-methyl	1	1	1	3	5	n.d.
parathion-methyl	1	1	1	3	5	n.d.
endosulfan	2	2	2	6	12	n.d.
fenpropimorph	< 3	< 3	< 3	< 3	< 6[a]	n.d.
fenpropimorph	< 3	< 3	< 3	< 3	< 6[a]	n.d.
lindane[b] [4]	2	3	4	26[c]	31[d]	n.d.
lindane[e] [4]	1	2	6	21[c]	23[d]	n.d.
trifluralin[b] [7]	14	14	12	23	64	n.d.
plant tests:						
parathion-methyl[f]	9	14	12	30	64	93 %
endosulfan[f]	12	15	13	21	60	89 %
fenpropimorph[f]	14	18	15	19	66	95 %
lindane [4]	4	8	12[g]	37[h]	55[d]	n.d.
trifluralin[f]	48	31	14	5	99	106 %

n.d., not determined; a, 4 times half of the detection limit of 3 % of each sampling interval; b, two bowls, c, 6 - 29 h sampling; d, interpolated data; e, six bowls; f, for these tests the volatilisation was calculated in relation to the a.i.-amount recovered by air sampling and from the plants; g, 3 - 7 h sampling; h, 7 - 29 h sampling.

The soil volatility of fenpropimorph was for all sampling intervals below the limit of detection corresponding to a volatilisation rate of < 6 % within 24 h. This result is confirmed by data of Ortmann [8], where under similar conditions a volatility of 7 % was found using ^{14}C-labelled

fenpropimorph. Ortmann [8] also performed two soil volatilisation tests with ^{14}C-labelled parathion-methyl. The volatility was determined to be 3 % for both tests. This is in relative good agreement with the 5 % volatility found for parathion-methyl in the present study.

For four plant tests a mass balance was calculated by summarizing application losses, recoveries from the plants, and the volatilised a.i.-amount. The mass balance accounts not for non-extractable pesticide residues (bound residues). However, it is assumed that this amount is low because of the relatively short duration of the tests (24 h). The recoveries for the tests were sufficient (mean value 96 %).

The volatilisation from plant surfaces was in the range 50 - 70 % for parathion-methyl, endosulfan, fenpropimorph, and lindane. Trifluralin was nearly completely volatilised within 24 h (99 %).

Using the same wind tunnel system and similar climatic conditions, Ortmann [8] found for two volatilisation tests with a ^{14}C-labelled parathion-methyl EC formulation plant volatilities of 77 % and 72 % within 24 h (recoveries 97 % and 93 %). Kubiak et al. [13] carried out comparative plant volatilisation experiments with parathion-methyl in the field and in a laboratory test system. For the laboratory test the climatic conditions of the field test were reproduced. The field volatilisation test with a wettable powder (WP) formulation of parathion-methyl yielded a volatilisation of 74.7 % within 24 h (determined indirectly by residue analysis). The respective laboratory test showed a volatilisation of 77.2 % after 24 h (determined by air sampling). The recovery for this laboratory experiment using ^{14}C-labelled parathion-methyl was 97.5 % (including 3 % bound residues). A further study by Stork et al. [14] revealed a plant volatility of 50 % for parathion-methyl within 24 h (also for a WP formulation). The results of all tests are not fully comparable because of differences of test performance, use of ^{14}C-tracer methods, formulation type and climatic conditions (temperature, air humidity, and air velocity). However, it seems that the use of non-labelled compounds for volatility testing is appropriate. This may be an advantage if formulations have to be tested which are difficult to prepare with ^{14}C-labelled compounds (e.g., SC, WP).

For all five compounds of this study volatilisation from French beans was higher as compared to soil surfaces. The plant volatility for parathion-methyl, endosulfan, and fenpropimorph was by factors of 5 - 13 higher than the respective soil volatility. For lindane and trifluralin the plant volatility was only by factors of 2 and 1.5, respectively, higher. If only the trifluralin volatilisation within the first hour is compared, a factor of 3 is found between soil and plant volatility (14 % versus 48 %).

The fact that plant volatility is always significantly higher than soil volatility also was reported for other compounds: e.g., triallate (Pestemer and Krasel [9]), diuron (Fritz et al. [10]), lambda-cyhalothrin, fluazifop-P-butyl, pirimicarb, prosulfocarb (Heath et al. [11]). The higher volatilisation rates from plants as compared to soil surfaces are caused probably by a higher air velocity directly above the leaves and a more turbulent air flow due to the more complex surface of a plant

stand. Volatilisation from soil surfaces is controlled mainly by diffusion of the pesticide through the stagnant boundary layer above the soil [12]. From these results it may be concluded that the measurement of plant volatility for most compounds is sufficient for the assessment of the volatility potential. Additional soil volatility tests may only be necessary for compounds used for soil treatments with a plant volatility clearly above the 20 % threshold of the BBA-guideline [1] (e.g., a volatilisation rate of > 40 % within 24 h).

In table 2 several volatilisation data from the soil and plant tests are listed as well as the vapour pressures. The sum of the volatilisation expressed as % of the initial amount seemed to be no good base for a comparison. Especially at higher volatilisation rates the surface becomes depleted of the pesticide and the volatilisation decreases. The highest volatilisation rates (expressed as % volatilised per hour) were always found during the first hour when the target area was still saturated with the applied pesticide. Therefore, the volatilisation of this first sampling period was assumed to be a more adequate measure of the volatilisation potential. In general, the volatilisation kinetics for all compounds were comparable (refer to the volatilisation data for the different sampling intervals in table 1).

Table 2:   Comparison of vapour pressure and volatilisation data for the first sampling interval (0 - 1 h-sampling) of soil and plant tests.

Test substance	vapour pressure at 20°C mPa	volatilisation within 1 h % of initial	1 h-average air concentration $\mu g/m^3$	1 h-mass flow mg/m^2
soil tests:				
parathion-methyl	0,2 [15]	1[a]	0.04[a]	0.21[a]
endosulfan	0,83 [16]	2	0.25	0.69
fenpropimorph	2,3 [15]	< 3[a]	< 0.5[a]	< 0.7[a]
lindane	5,6 [15]	1[a]	0.26[a]	0.81[a]
trifluralin	13,7[b] [15]	14	0.94	9.6a
plant tests:				
parathion-methyl	0,2 [15]	9	0.29	0.8
endosulfan	0,83 [16]	12	0.77	2.4
fenpropimorph	2,3 [15]	14	1.5	4.3
lindane	5,6 [15]	4	0.84	2.2
trifluralin	13,7[b] [15]	48	2.6	6.9

a, mean value of two tests; b, at 25°C

When discussing the data one has to assume that vapour pressure data are difficult to determine. Different methods are yielding differing values for the same substance (e.g. for lindane data in the range from 1.24 to 15 mPa at 20°C [17]). Therefore, the correlation depends on the quality of the vapour pressure data.

The pesticides tested here were in the vapour pressure range 0.2 - 13.7 mPa. With the exception of the soil volatilisation tests with fenpropimorph, all pesticides were clearly volatile under the conditions applied. Chemicals with low vapour pressures have been found to be non-volatile under similar conditions [3]. Examples are deltamethrin (vapour pressure $2 * 10^{-3}$ mPa at 25°C [15]), simazin (vapour pressure $8.1 * 10^{-4}$ mPa at 20°C [15]), and di-(2-ethyl hexyl) phthalic acid ester (DEHP), a compound widely used as plasticizer (vapour pressure $6 * 10^{-3}$ mPa at 20°C [18]).

For the evaluation of the volatilisation data of the five pesticides different correlations were analysed (separately for soil and plant volatilisation). For this data set, correlation coefficients ≥ 0.88 are statistically significant with a probability of ≥ 95 %. A correlation coefficient of $r = 0.79$ was found for the relation between the averaged pesticide air concentrations during the first hour of the soil tests and the vapour pressure (respective correlation for plant volatilisation: $r = 0.87$). The correlation between the first hour mass flow and soil volatilisation was $r = 0.93$ (plant volatilisation: $r = 0.84$). The compound which showed a lower volatility than expected was lindane. This may be caused by the fact that lindane was used as suspension concentrate formulation, while for the other compounds emulsion concentrate formulations were used. In an emulsion the active ingredient is completely dissolved and is evenly distributed on the treated surface after application while in a suspension the pesticide distribution on the surface is probably more uneven.

The respective correlations data for the four EC applied pesticides without lindane were: correlation vapour pressure - 1 h-air concentration soil tests: $r = 0.88$, correlation vapour pressure - 1 h-air concentration plant tests: $r = 0.93$, correlation vapour pressure - 1 h-mass flow soil volatilisation: $r = 0.99$, correlation vapour pressure - 1 h-mass flow plant volatilisation: $r = 0.90$ (correlation coefficients ≥ 0.96 are statistically significant with a probability of ≥ 95 %).

Although it is not possible to use the correlation to calculate the exact volatilisation value according to the BBA-guideline [1] (i.e., as % volatililised in relation to the initial dose) the data suggest that a ranking of pesticides in respect to their volatilisation potential is possible. Compounds with vapour pressures in the mPa-range probably yield volatilisation of above 20 % in soil tests (threshold criteria of the BBA-guideline for further testing [1]). A respective critical vapour pressure for plant test can not be derived because all model compounds of the set showed volatilisation rates clearly above 50 %.

CONCLUSIONS

The present study revealed that pesticide volatility testing according to the BBA-guideline [1] with non-labelled compounds is feasible. Earlier studies mainly were performed with ^{14}C-labelled compounds (e.g., [3], [10], [11], [13], [14]). However, Dörfler et al. used both, labelled and non-labelled test substances, for volatility studies in a laboratory model system [19]. Due to lower safety regulations the use of non-labelled compounds is preferred if the analytical method is sensitive enough. As further advantage, compounds may be used directly in the commercial formulations (no special preparation of ^{14}C-labelled formulations is necessary).

Data from this study and further data from different authors suggest that plant volatility is always higher than soil volatility. Therefore, it may be appropriate to omit the volatilisation tests with bare soil and only perform plant volatilisation tests. In most cases these results will be sufficient to assess the volatilisation behaviour of pesticides.

The data presented here reveal that a correlation between volatilisation and vapour pressure is likely. Experiments with a further set of pesticides with different vapour pressure especially in the relevant range 10^{-3} - 10^{-4} Pa are necessary to confirm these results. Additionally, the influence of the formulation type on the volatilisation behaviour of pesticides should be tested.

A large number of volatility tests especially with low vapour pressure compounds can be avoided in the future if low volatility pesticides could be identified by their vapour pressure.

ACKNOWLEDGEMENT

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